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IS 12540 (1988): Acrylonitrile [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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Indian Standard

SPECIFICATION FOR ACRYLONITRILE

UDC 661·7 : 547·339·2'361·2

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 7

June 1989

Indian Standard
SPECIFICATION FOR ACRYLONITRILE

0. FOREWORD

0.1 This Indian Standard was adopted by the Bureau of Indian Standards on 29 July 1988, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Acrylonitrile ($CH_2 = CH\text{-}C{\equiv}N$) is a highly toxic mobile colourless liquid. It is also known as propenenitrile and vinyl cyanide. It is used in the manufacture of synthetic rubber, plastics, synthetic fibres, etc. Acrylonitrile also finds wide applications in organic synthesis and grain fumigants.

0.3 In the preparation of this standard, assistance has been derived from GOST 11097-1964 'Acrylonitrile, commercial grade', issued by the Committee on Standards, Measures and Measuring Instruments of the USSR.

0.4 The Committee at the time of finalization of the draft envisaged to incorporate the requirement of acrylonitrile at a later date to cater to the need of fibre industry specifically after the necessary details for the same are made available.

0.5 Code of safety for handling this chemical has been separately given in IS : 11141-1984*.

0.6 Clause 2.2.1 of this standard calls for an agreement between the purchaser and the supplier regarding the use of an alternate stabilizer.

0.7 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the results of a test or analysis, shall be rounded off in accordance with IS : 2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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*Code of safety for acrylonitrile.

†Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements, methods of sampling and test for acrylonitrile.

2. REQUIREMENTS

2.1 The material shall be colourless liquid free from suspended matter. The observation of the colour shall be made in the transmitted light (*see* **2.2.1**).

2.2 The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods prescribed in Appendix A. Reference to relevant clauses of the appendix are given in col 4 of the table.

2.2.1 On agreement between the purchaser and the supplier, hydroquinone in acrylonitrile equivalent to 0·1 percent of the mass may be used for stabilizing the product instead of monoethyl ether of hydroquinone (MEHQ). In this case, the product may have a yellow tint. The amount of hydroquinone added shall be guaranteed by the manufacturer. A method of test for determination of hydroquinone content has been given in Appendix B for guidance purposes.

2.2.2 The acetonitrile content shall not be determined in acrylonitrile obtained from hydrocyanic acid and acetylene.

3. PRECAUTIONS IN STORING AND HANDLING

3.1 As acrylonitrile is flammable, necessary safeguards against the risk arising from storage and handling of large volumes of flammable liquids shall be provided and all due precautions taken at all times to prevent accident by fire or explosion. It shall be stored in a cool place. Open flames and smoking shall not be allowed where it is stored or handled. Storage vessels shall be vented to safe atmosphere.

3.2 Except when they are opened for the purpose of cleaning and rendering them free from acrylonitrile vapours, all empty tanks and other containers shall be securely closed (*see* **0.5**).

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in mild steel cans and drums. For transportation purposes, it shall be filled in tank cars and tank trucks.

TABLE 1 REQUIREMENTS FOR ACRYLONITRILE

(*Clause* 2.2)

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CLAUSE NO. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Distillation range at 760 mm Hg:		
	a) Initial boiling point, *Min*	74·5°C	A-2
	b) Dry point, *Max*	79·0°C	
ii)	Assay, percent by mass, *Min*	99·0	A-3
iii)	Water content, percent by mass, *Max*	0·8	A-4
iv)	Specific gravity, at 27°C/27°C	0·800 to 0·802	A-5
v)	Refractive index at 27°C	1·388 2 to 1·389 2	A-6
vi)	Hydrocyanic acid content, percent by mass, *Max*	0·001	A-7
vii)	Soluble iron content, percent by mass, *Max*	0·000 01	A-8
viii)	Methyl vinyl ketone, percent by mass, *Max*	0·03	A-9
ix)	*Acetonitrile, percent by mass, *Max*	0·05	A-10
x)	Acetaldehyde content, percent by mass, *Max*	0·005	A-11
xi)	Ammonia content, percent by mass, *Max*	0·07	A-12
xii)	Monoethyl ether of hydroquinone (MEHQ), ppm, *Max*	45	A-13
xiii)	*p*H (5 percent aqueous solution)	6 to 7·5	A-14
xiv)	Colour, APHA, *Max*	15	A-15
xv)	Non-volatile matter, percent by mass, *Max*	0·01	A-16
xvi)	Peroxide content, percent by mass, *Max*	0·000 02	A-17
xvii)	Heavy metals (as Cu), percent by mass, *Max*	0·000 01	A-18

*See 2.2.2.

4.2 Marking — Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;

b) Net mass of the material in the container;

c) Name of the manufacturer and his recognized trade-mark, if any;

d) Lot or batch number in code or otherwise; and

e) The symbol given in Fig. 5 of IS : 1260 (Part 1)-1973* and the words 'HARMFUL VAPOUR, DO NOT INHALE, FLAMMABLE, KEEP IN COOL PLACE'.

4.2.1 The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5. SAMPLING

5.1 The procedure for sampling and criteria for conformity of the material shall be as prescribed in Appendix C.

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*Pictorial markings for handling and labelling of goods: Part 1 Dangerous goods (*first revision*).

APPENDIX A

(*Clause* 2.2 and *Table* 1)

METHODS OF TEST FOR ACRYLONITRILE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF DISTILLATION RANGE

A-2.1 Determine the distillation range following the procedure given in IS : 5298-1983†.

A-3. ASSAY

A-3.1 Apparatus

A-3.1.1 *Conical Flasks* — (*see* Fig. 1).

A-3.1.2 *Pipette* — with a mark at 1·1 to 1·5 ml.

A-3.2 Reagents

A-3.2.1 *Sodium Sulphite Anhydrous Solution* — 2 N.

A-3.2.2 *Standard Sulphuric Acid Solution* — 1 N.

A-3.2.3 *Thymolphthalein Alcoholic Solution* — 0·1 percent.

A-3.3 Procedure — In order to take the weighed portion, fill flask *B* of the apparatus (*see* Fig. 1) with the product under test and then fill the pipette of the apparatus with it to the mark. Remove the pipette, wipe it with filter paper, insert it in flask *A* and weigh to the nearest 0·000 2 g. Transfer the contents of the pipette to a 250-ml conical flask provided with a ground-glass stopper, into which 50 ml of neutral sodium sulphite solution and 25 ml of water have already been added. Quickly

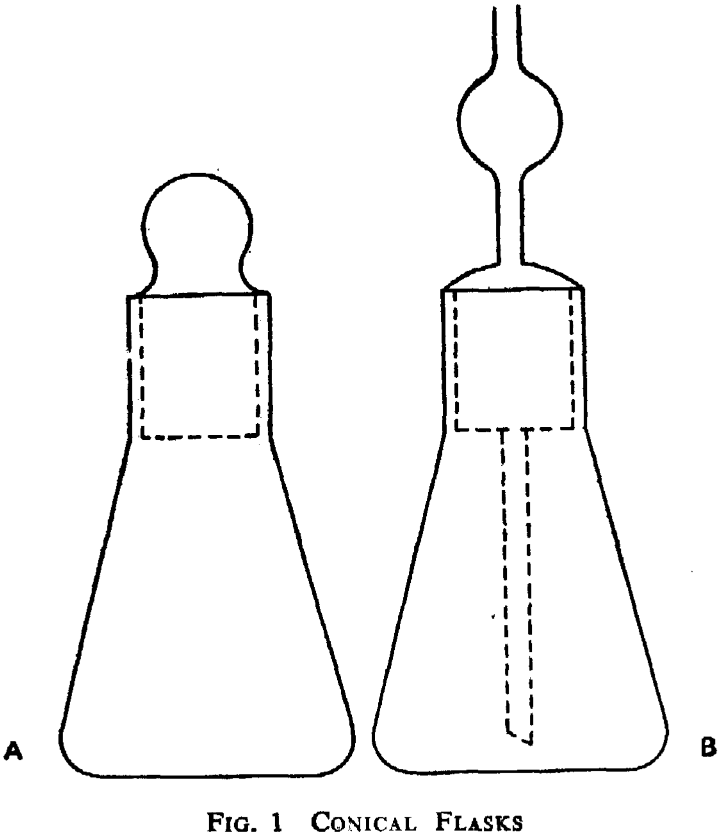

FIG. 1 CONICAL FLASKS

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*Specification for water for general laboratory use (*second revision*).

†Method for the determination of distillation range and of distillation yield (*first revision*).

close the flask with its stopper and keep it for 3 minutes in warm water (30 to 35°C), shaking it gently from time to time without removing it from water. Immediately insert the pipette into the neck of the flask *A* and reweigh with the same accuracy. The weighed portion shall be found from the difference between the two weighings. The weighed portion shall not be more than 1·2 g. After three minutes have elapsed, remove the flask from the water, shake it up for 1 minute, add 6 or 7 drops of the thymolphthalein solution and titrate the solution under test with standard sulphuric acid solution until blue colour disappears.

A-3.4 Calculation

A-3.4.1 The content of acrylonitrile in percent by mass shall be calculated from the formula:

$$\text{Acrylonitrile content, percent by mass} = \frac{V \times 0{\cdot}053 \times 100}{M}$$

where

V = volume in ml of standard sulphuric acid solution (1 N) used in the titration,

0·053 = amount in g of acrylonitrile corresponding to 1 ml of standard sulphuric acid solution (1N), and

M = mass in g of the material taken for the test.

NOTE 1 — A blank test shall be carried out under the test conditions specified above in order to check the sodium sulphite for neutrality. The solution should remain colourless. If the sulphite solution reveals a slightly alkaline reaction, it may be neutralized with a calculated amount of acid and then checked for neutrality to thymolphthalein.

NOTE 2 — The assay may also be determined by deducting the sum of percent content of impurities from 100.

A-4. DETERMINATION OF WATER CONTENT

A-4.1 Take 0·5 to 1·5 g weighed portion with the aid of the apparatus described in **A-3.1**. Determine the moisture content by the use of Karl Fischer method as given in IS : 2362-1973*.

A-5. DETERMINATION OF SPECIFIC GRAVITY

A-5.1 Apparatus

A-5.1.1 *Pyknometer or Specific Gravity Bottle* — 25 ml capacity.

A-5.1.2 *Water-Bath* — maintained at 27·0 ± 0·2°C.

A-5.2 Procedure — Clean and dry the pyknometer or specific gravity bottle. Weigh it, fill it with freshly boiled distilled water, place it in the bath maintained at 27·0±0·2°C and allow sufficient time (about 45 minutes) to attain the temperature of the bath. Then insert the capillary stopper which has been brought to 27·0±0·2°C. Wipe the excess liquid from the stopper, remove the pyknometer or the specific gravity bottle from the bath, bring to room temperature and weigh. Empty the pyknometer or the specific gravity bottle, clean and dry it, and repeat the operation with the material at 27·0±0·2°C.

A-5.3 Calculation

$$\text{Specific gravity at } 27^\circ\text{C}/27^\circ\text{C} = \frac{A - B}{C - B}$$

where

A = mass in g of the pyknometer or specific gravity bottle filled with the material,

B = mass in g of the dry pyknometer or specific gravity bottle, and

C = mass in g of the pyknometer or specific gravity bottle filled with water.

A-6. DETERMINATION OF REFRACTIVE INDEX

A-6.0 Refractive Index — The ratio of the sine of angle of incidence to the sine of angle of refraction when a ray of light of defined wavelength passes from air into the material kept at constant temperature is the refractive index. The wavelength is 589·3±0·3 nm corresponding with D_1 and D_2 lines of sodium spectrum. The reference temperature shall be 27°C except for those which are not liquid at this temperature, in which case a higher temperature (say 30°C) depending on the melting point of the material shall be used.

A-6.1 Apparatus

A-6.1.1 *Refractometer* — A recognized type of refractometer allowing direct readings of refractive indices between 1·300 0 and 1·700 0 with an accuracy of ±0·000 2.

A-6.1.2 *Apparatus for Temperature Maintenance* — Any apparatus which ensures a circulation of water through the refractometer and maintains the temperature within ±0·2°C.

A-6.1.3 *Light Source* — Sodium light. Diffused day light or light from an electric lamp may be used for instruments fitted with an achromatic compensator.

A-6.2 Procedure — Pass a stream of water through the instrument so as to keep it at the temperature

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*Determination of water by the Karl Fischer method (*first revision*).

at which the readings are required to be taken. This temperature shall not differ from the reference temperature by more than ±0·2°C. Before placing the material in the instrument, bring the temperature of test sample to that at which the measurement is made. Take readings only when the temperature is stable.

NOTE — Since moisture in the air may condense in the cooled prisms, great care shall be exercised when determining refractive indices during hot and humid weather.

A-6.3 Calculation

A-6.3.1 The refractive index n_d^t at the specified temperature is given by the following formula:

$$n_d^t = n_d^t + 0{\cdot}000\,4\,(t' - t)$$

where $n_d^{t'}$ is the reading taken at the working temperature t'. Express result to four decimal places.

NOTE — If, for any reason, it is not possible to determine the refractive index at the specified temperature, then the value of refractive index may be calculated by any applying suitable correction factor or a factor of 0·000 38 per degree celsius if the factor is not available.

A-7. DETERMINATION OF HYDROCYANIC ACID CONTENT

A-7.1 Apparatus

A-7.1.1 *Spectrophotometer or Photoelectric Colorimeter* — equipped with cells of 5 cm lightpath.

A-7.2 Reagents

A-7.2.1 *Copper Sulphate Solution* — 0·02 percent.

A-7.2.2 *Potassium Cyanide*

A-7.2.3 *Potassium Hydroxide Solution* — 0·03 percent.

A-7.2.4 *Rectified Spirit* — conforming to IS : 323-1959*.

A-7.2.5 *Buffer Solution* — To prepare the solution, dissolve 2·15 g of di-substituted sodium phosphate in 1 litre of distilled water.

A-7.2.6 *Acrylonitrile* — free from hydrocyanic acid.

A-7.2.7 *Phenolphthalein Solution* — Prepare the solution as follows:

Dissolve 37·5 g of sodium hydroxide in 750 ml of distilled water and add 25 g of phenolphthalein. Heat the resulting solution to a temperature of 70°C and, while stirring constantly, add 17·5 g of zinc dust. Heat the solution to boiling and continue the reaction until the solution is completely decolorized. Then cool the solution and add approximately 200 ml of distilled water and then hydrochloric acid until precipitation is complete. Separate the precipitate from the solution by decantation, filter it out on a Buchner funnel and wash it with distilled water until the wash water reacts neutral. Then dissolve the precipitate on the filter paper in hot alcohol. After that, add water to the alcoholic solution until a white deposit (phenolphthalein) is completely precipitated. Filter out the precipitate and dry it at a temperature of 110 to 120°C (its melting point is 225°C). If necessary, recrystallize. Dissolve the dry phenolphthalein in ethyl alcohol, taking 0·5 g per 100 ml of alcohol.

Introduce 3 ml of the alcoholic phenolphthalein solution into a 100-ml measuring flask and dilute the solution to the mark with the copper sulphate solution. The solution shall be prepared 1 hour before use.

A-7.3 Plotting the Calibration Curve — Prepare the solution for calibration as follows:

Weigh out 0·6 g of potassium cyanide to the nearest 0·000 3 g, transfer the weighed portion quantitatively to a 1 litre measuring flask, dissolve in water and dilute to the mark. Pipette 5 ml of the resulting solution into a 100-ml measuring flask and dilute with freshly boiled water to the mark (Solution A).

The content of potassium cyanide in 1 ml of Solution A corresponds to a 0·001 2 percent hydrocyanic acid solution which is equivalent to its content calculated for a 3 ml sample of acrylonitrile equal to 0·000 5 percent.

To plot the calibration curve, pipette 3·0 ml of acrylonitrile free from hydrocyanic acid into five 50 ml measuring flasks, each containing 20 ml of freshly boiled water and add to each flask 1·0, 2·0, 3·0, 4·0 and 5·0 ml of Solution A, respectively. Then add to each flask, 10 ml of the buffer solution, 5 ml of the phenolphthalein solution and mix the contents of the flasks thoroughly. Now add 5 ml of the potassium hydroxide solution to each flask, dilute the solution in each flask with water to the mark and mix.

Introduce the solution under test into a cell, the height of the liquid being 10 mm, and determine the optical density at a wavelength of 554 nm. Fill a second cell with distilled water for comparison.

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*Specification for rectified spirit (*revised*).

The values obtained shall be plotted with the optical density as ordinates and the percentage of hydrocyanic acid as abscissas.

A-7.4 Procedure — Introduce 3·0 ml of the material into a 50-ml measuring flask containing 20 ml of freshly boiled water. Add to flask, 10 ml of the buffer solution (**A-7.2.5**), 5 ml of phenolphthalein solution (**A-7.2.7**) and mix the contents of the flask thoroughly. Now add 5 ml of potassium hydroxide solution (**A-7.2.3**) to the flask, dilute the solution with water to the mark and mix.

A-7.4.1 Introduce the solution under test into cell, the height of the liquid being 10 mm and determine the optical density at a wavelength of 554 nm. Fill a second cell with distilled water for comparison.

A-7.4.2 The solutions shall be tested in the spectrophotometer immediately after they are prepared.

A-7.5 Calculation — The content of hydrocyanic acid in percent shall be found by dropping a perpendicular from the point of intersection of the horizontal line of the measured optical density and the calibration curve.

A-8. DETERMINATION OF SOLUBLE IRON CONTENT

A-8.1 Apparatus

A-8.1.1 *Spectrophotometer*

A-8.2 Reagents

A-8.2.1 *Hydrochloric Acid Solution* — 1 : 1.

A-8.2.2 *Hydroxylamine Hydrochloride Solution* — 10 percent.

A-8.2.3 *Orthophenanthroline Solution* — 0·1 percent.

A-8.2.4 *Standard Iron Solution* — 1 ml of solution shall contain 0·1 mg of iron.

A-8.2.5 *Aqueous Ammonia*

A-8.3 Plotting the Calibration Curve — Using a burette, introduce 0·5, 1·0, 1·5, 2·0 and 3·0 ml of the solution containing iron, respectively, into 100 ml measuring flasks. Then add 9 ml of the hydroxylamine hydrochloride solution to each flask and insert a strip (1 cm wide) of Congo red indicator paper in each solution.

Neutralize the solutions with aqueous ammonia until the indicator paper turns red. Further, add 10 ml of orthophenanthroline solution to each flask and dilute the solution with water to the mark. Mix the solution thoroughly.

Determine the optical density of the resulting solution in spectrophotometer at a wavelength 510 nm, the height of the solution being 10 mm. The solution taken for comparison shall be distilled water.

The test results obtained shall be plotted with the optical density as ordinates and the content of iron in micrograms in 5 ml of the solution as abscissas.

A-8.4 Procedure — Filter the sample under test through a glass funnel with a No. 3 porous plate to free it from mechanical impurities. Evaporate 100 ml of the clear filtrate to dryness in a quartz dish on a steam bath. Place the dish in a muffle furnace and keep it there at a temperature of 500 to 550°C for 15 minutes. After cooling, add 1 ml of the hydrochloric acid solution to the dry residue, partly cover the dish with a watch glass and evaporate on a steam bath for 30 minutes. Then wash the glass with water allowing the wash water to drain into the dish, and evaporate until one drop remains. Add 1 ml of the hydroxylamine hydrochloride solution (**A-8.2.2**) into the dish, insert a strip of the Congo red indicator paper, add aqueous ammonia (1 or 2 drops) until the indicator paper turns red. Further, add to the dish, 1 ml each of water and orthophenanthroline solution (**A-8.2.3**), transfer the solution to a 5-ml measuring flask, dilute the solution with water to the mark and mix.

Determine the optical density of the resulting solution in a spectrophotometer at a wavelength 510 nm, the height of the solution being 10 mm. Distilled water shall be taken as the reference solution. By dropping a perpendicular from the point of intersection of the horizontal line of the measured optical density and the calibration curve, find the iron content in micrograms in 5 ml of the solution.

A-8.5 Calculation — The content of soluble iron in percent mass shall be calculated from the formula:

$$\text{Soluble iron content, percent by mass} = \frac{C \times 100}{0{\cdot}8 \times V \times 1\,000\,000}$$

where

C = amount of iron in micrograms in 5 ml of the solution found from the calibration curve,

V = volume in ml of acrylonitrile taken for the test, and

0·8 = density of the acrylonitrile.

A-9. DETERMINATION OF METHYL VINYL KETONE CONTENT

A-9.1 Apparatus — Infrared spectrophotometer with sodium chloride cell of 0·5 mm path.

A-9.2 Reagents

A-9.2.1 *Acrylonitrile* — free from methyl vinyl ketone.

A-9.2.2 *Methyl Vinyl Ketone*

A-9.3 Plotting the Calibration Curve — To plot the calibration curve, prepare solutions of acrylonitrile containing 0·05, 0·1, 0·2, 0·3 and 0·4 percent of methyl vinyl ketone.

Take the spectrum for the resulting solutions in the range of wavelengths of 7·6 to 9·0 μ and determine the optical density of the 8·44 μ analysis line of the solution in the sodium chloride cell.

The values obtained shall be plotted against the concentration of methyl vinyl ketone in percent as abscissas and the optical density as ordinates.

A-9.4 Procedure — Introduce the material under test into the cell. Take the spectrum in the range of wavelength of 7·6 to 9·0 μ and determine the optical density of the 8·44 μ analysis line. The amount of methyl vinyl ketone shall be found by dropping a perpendicular from the point of intersection of the horizontal line of the optical density obtained and the calibration curve.

A-10. DETERMINATION OF ACETONITRILE CONTENT

A-10.1 Apparatus — Chromatograph with ionization detector.

A-10.2 Reagents

A-10.2.1 *Diethyldithiocyanide* — boiling point 178 to 179°C at 3 mm Hg.

A-10.2.2 *Inza Crushed Firebrick* — grade 600, purified.

A-10.2.3 *Nitrogen Gas*

A-10.2.4 *Hydrozen* — commercial grade.

A-10.2.5 *Chloroform* — (*see* IS : 5296-1969*).

A-10.3 Procedure — When carrying out the test on the chromatograph, the following conditions shall be observed:

a) The fixed phase shall be diethyldithiocyanide.

b) The inert solid support shall be purified in crushed fire-brick, grade 600, with a particle size of 0·25 to 0·5 mm.

c) The ratio of the mass of the fixed phase to the mass of the support shall be 15 : 85.

d) The column length shall be 1·6 m.

e) The column diameter shall be 4 mm.

f) The temperature in the thermostat shall be +50°C.

g) The carrier gas shall be nitrogen with a gas velocity of 30 cm^3 per minute at a temperature of 20°C and a pressure of 760 mm Hg.

h) The volume of the sample taken for the test shall be approximately 0·01 ml.

A-10.3.1 The column packing shall be prepared as follows:

Pour a 10 percent solution of hydrochloric acid on the sifted 0·25 to 0·5 mm fraction of crushed firebrick, allow it to stand for one hour, then pour off the acid, wash the crushed firebrick 10 to 15 times with water until free from chlorides and dry in a drying chamber at a temperature of 140 to 150°C for 3 to 4 hours.

Take 20 g of the purified crushed firebrick and cover it with the solution of diethyldithiocyanide in chloroform or alcohol (3·55 g of diethyldithiocyanide in 50 to 60 ml of the solution) and dry in a drying chamber at a temperature of 100°C for 3 to 4 hours to constant mass.

The content of acetonitrile in the acrylonitrile shall be determined from the chromatogram obtained by partition chromatography.

A-10.4 Calculation — Preliminarily, from chromatograms of specially prepared mixtures with known concentrations of acetonitrile and acrylonitrile, calculate the calibration factor of the peak area of acetonitrile with respect to the peak area of acrylonitrile (K_{ac}) from the formula:

$$K_{ac} = \frac{C_1 S_0}{C_0 S_1}$$

where

S_0 = peak area of the acrylonitrile (the calibration factor of the peak area shall be taken to be 1),

S_1 = peak area of the acetonitrile,

C_1 = concentration of acetonitrile in the known mixture, and

C_0 = concentration of acrylonitrile in the mixture.

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*Specification for chloroform, technical and analytical (*revised*).

The content of acetonitrile in the acrylonitrile under test in percent (C_{ac}) shall be calculated from the formula:

$$C_{ac} = \frac{K_{ac} \times S_{ac} \times 100}{S_{acr}}$$

where

S_{ac} = peak area of the acetonitrile, and

S_{acr} = peak area of the acrylonitrile.

A typical section of a chromatogram for acetonitrile and acrylonitrile is shown in Fig. 2.

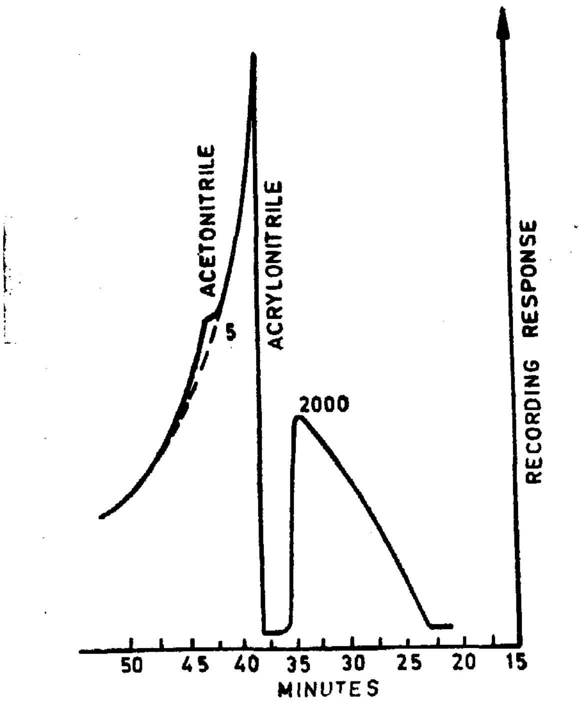

FIG. 2 CHROMATOGRAM FOR ACETONITRILE AND ACRYLONITRILE

The peak area shall be determined by weighing the corresponding section of the chromatogram to the nearest 0·000 2 g.

The average error in determination should not exceed 20 percent (relative). The smallest concentration of acetonitrile that can be determined is approximately 0·007 percent.

A-11. DETERMINATION OF ACETALDEHYDE

A-11.1 Apparatus — Spectrophotometer.

A-11.2 Reagents

A-11.2.2 *Schiff's Reagents* — prepared by mixing equal volumes of Solution A and Solution B as below:

a) *Solution A* — 0·1 percent solution of Fuchsin in water.

b) *Solution B* — prepared by introducing 6·5 g of sodium pyrosulphate into a 100-ml measuring flask containing 65 ml of a 1 N solution of hydrochloric acid and diluting the resulting solution with distilled water to the mark.

Cool Solution A and Solution B to a temperature of 10°C, combine them and allow the mixture to stand at a temperature not exceeding 15°C until it is completely decolorized. Then filter the Schiff's reagent and keep the filtrate at a temperature not exceeding 15°C in a brown glass bottle.

A-11.2.2 *Acrylonitrile* — free from acetaldehyde.

A-11.3 Plotting the Calibration Curve — In order to plot the calibration curve, prepare solutions of acetaldehyde in acrylonitrile, having the concentrations as 0·05, 0·02, 0·01, 0·005 and 0·001 percent.

Pipette 20 ml of each solution to separate 25-ml measuring flasks, then add 2 ml of Schiff's reagent to each flask and dilute each solution to the mark with water. After allowing the solution to stand for 1 hour at a temperature of 80°C, determine the optical density of each solution at a wavelength of 590 nm, the height of the solution in the cell being 10 mm.

The values obtained shall be plotted with optical density as ordinates and the concentration of acetaldehyde in percent as abscissas.

A-11.4 Procedure — Pipette 20 ml of the sample under test into a 25-ml measuring flask, and 2 ml of Schiff's reagent and add water to the mark. After allowing the solution to stand for 1 hour at a temperature of 8°C, measure the optical density at a wavelength of 190 nm, the height of the solution in the cell being 10 mm.

The percentage of acetaldehyde in the sample of acrylonitrile under test shall be found with the aid of the calibration curve from the optical density value obtained.

A-12. DETERMINATION OF AMMONIA CONTENT

A-12.1 Reagents

A-12.1.1 *Standard Hydrochloric Acid Solution* — 0·01 N.

A-12.1.2 *Methyl Orange Aqueous Solution* — 0·1 percent.

A-12.2 Procedure — Introduce 20 ml of water and 1·0 ml (0·8 g) of the acrylonitrile under test into a 100 ml conical flask provided with a ground glass stopper. Close the flask tightly and mix the solution thoroughly. Then add 2 to 3 drops of methyl orange solution to the contents of the flask and titrate from a microburette with the standard hydrochloric acid solution until the colour of the solution changes from yellow to orange-red.

A-12.3 Calculation — The content of amines, expressed as ammonia, in percent shall be calculated from the formula:

$$\text{Ammonia content, percent by mass} = \frac{V \times 0{\cdot}000\ 17 \times 100}{M}$$

where

V = volume in ml of standard hydrochloric acid solution (0·01 N) used in the titration,

M = mass in g of the acrylonitrile taken for the test, and

0·000 17 = mass in g of ammonia corresponding to 1 ml of standard hydrochloric acid solution (0·01 N).

A-13. DETERMINATION OF MONOMETHYL ETHER OF HYDROQUINONE (MEHQ)

A-13.1 Apparatus

A-13.1.1 *UV Spectrophotometer*

A-13.1.2 *Measuring Flasks* — 50 ml.

A-13.1.3 *Pipette* — 5 ml.

A-13.2 Reagents

A-13.2.1 *Glacial Acetic Acid*

A-13.2.2 *Saturated Sodium Nitrite Solution*

A-13.3 Procedure — Pipette 5 ml of the sample in 50 ml measuring flask, add 10 ml of glacial acetic acid using a pipette and further add 0·5 ml of saturated sodium nitrite solution. Shake the flask to mix well and allow to stand exactly for one minute and dilute with water to 50 ml.

A-13.3.1 After 15 minutes, measure the extinction on UV spectrophotometer at 420 nm of the solution above, using 10 mm cell, and compare with that of the blank solution which is prepared by mixing 10 ml of glacial acetic acid, 0·5 ml of saturated sodium nitrite solution and water added to make it a total of 50 ml.

A-13.4 Calculation — Calculate the monomethyl ether of hydroquinone (MEHQ) content by the following formula:

$$\text{Monomethyl ether of hydroquinone content (MEHQ), ppm} = \frac{M_1}{M_2}$$

where

M_1 = mass in g of MEHQ determined by the calibration curve, and

M_2 = mass of the sample in g (5 ml × 0·8).

A-14. DETERMINATION OF *p*H (5 PERCENT AQUEOUS SOLUTION)

A-14.1 Apparatus

A-14.1.1 *p*H *Meter*

A-14.1.2 *Magnetic Stirrer with Glass Stirring Bars*

A-14.1.3 *Beaker* — 100-ml.

A-14.1.4 *Pipette* — 5 ml capacity.

A-14.2 Reagents

A-14.2.1 *Reference Buffer Solutions* (Standards *p*H 4 and 7)

A-14.3 Procedure — Transfer 76 ml of distilled water to 100 ml glass beaker. Using 5-ml pipette, transfer 5-ml sample to above beaker. Place the beaker on magnetic stirrer with glass stirring bar. Allow acrylonitrile to dissolve. Immerse the electrode of *p*H meter in the above solution. Avoid the entrainment of any bubbles. Measure *p*H on *p*H meter. Calibrate the *p*H meter with the standard buffer solutions before actual measurement of *p*H of solution.

A-15. DETERMINATION OF COLOUR APHA

A-15.1 Apparatus

A-15.1.1 *Colour Comparison Tube Support* — permitting visual comparison through tall-form, 100 ml Nessler tubes in the direction of their longitudinal axis. It should be so constructed that white light is passed through or reflected off a white glass plate and directed with equal intensity through the tubes and should be shielded so that no light enters the tube from the side.

A-15.1.2 *Nessler Tubes for Colour Comparison* — tall form matched, 100-ml capacity.

A-15.2 Reagent

A-15.2.1 *Platinum Cobalt Stock Solution* — Dissolve 1·245 g of potassium chloroplatinate ($K_2P_1Cl_8$) and 1·000 g of cobaltous chloride ($C_0Cl_2 \cdot 6H_2O$) in water. Carefully, add 100 ml of hydrochloric acid (sp gr 1·19) and dilute to 1 000 ml with water.

A-15.2.1.1 Dilute 0, 1, 2, 3 and 4 ml of platinum cobalt stock solution (**A-15.2.1**) in separate 100 ml Nessler tubes up to the mark with water. Cap each tube. These standards should be freshly prepared.

A-15.3 Procedure — Fill, 100 ml of sample in 100-ml Nessler tube. Cap the tube. Place the sample tube in tube support and compare against standards. Look vertically downwards through the tubes towards the white glass plate of the tube support.

A-16. DETERMINATION OF NON-VOLATILE MATTER

A-16.1 Apparatus

A-16.1.1 *Evaporating Dish* — 200 ml capacity made of glass or porcelain.

A-16.1.2 *Warming Plate* — capable of maintaining a temperature of 80 ± 2°C. Heating element of plate should be enclosed so that acrylonitrile fumes cannot be ignited. The warming plate must be located in fume cupboard.

A-16.1.3 *Constant Temperature Oven* — capable of maintaining temperature of 105 ± 2°C.

A-16.1.4 *Desiccator*

A-16.1.5 *Pair of Tongs* — for handling evaporating dishes.

A-16.1.6 *Graduated Cylinder* — 100-ml.

A-16.2 Reagent

A-16.2.1 *Desiccant* — indicating type.

A-16.3 Procedure — Clean the evaporating dish and dry at 105 ± 2°C for 30 minutes. Remove the dish and cool it in desiccator for 30 minutes. Weigh the dish up to 0·1 mg accuracy. Measure 125 ml of sample by graduated cylinder and pour it in evaporating dish. Place the dish on warming plate at 80 ± 2°C. Allow the sample to evaporate in the fume cupboard. Place the dish in oven at 105 ± 2°C for 30 minutes. Remove the dish and place in desiccator for 30 minutes. Weigh the dish up to 0·1 mg accuracy.

A-16.4 Calculation

A-16.4.1 Calculate the concentration of non-volatile matter in percent (*m/m*).

$$\text{Non-volatile matter, percent by mass} = \frac{(M_2 - M_1) \times 100}{V \times 0{\cdot}8}$$

M_1 = mass of empty evaporating dish,

M_2 = mass of evaporating dish with non-volatile matter, and

V = volume of sample in ml.

A-17. DETERMINATION OF PEROXIDE CONTENT

A-17.1 Apparatus

A-17.1.1 *Erlenmeyer Flask, Glass Stoppered* — 125-ml capacity.

A-17.1.2 *Spectrophotometer* — visible range with 10 mm path cells.

A-17.2 Reagents

A-17.2.1 *Potassium Iodide* — reagent grade.

A-17.2.2 *Acetic Anhydride*

A-17.2.3 *Hydrogen Peroxide* — 30 percent CP.

A-17.2.4 *IRA-410 Ion-Exchange Resin*

A-17.3 Procedure — Pipette 25 ml each of sample and peroxide free acrylonitrile in two separate 125 ml glass stoppered Erlenmeyer flasks (peroxide free acrylonitrile can be prepared by passing acrylonitrile through a column of IRA-410 resin). Add 5 ml of acetic anhydride to each flask by pipette. Add 0·5 g of potassium iodide to each flask. Stopper the flask and swirl for one minute. Fill the two cells with blank and sample and find out absorbance exactly 20 minutes after addition of potassium iodide. Find out absorbance at 365 nm adjusting zero of the instrument by blank.

A-17.4 Preparation of Calibration Curve — Dilute 6·0 ml of 30 percent H_2O_2 to 100 ml with distilled water (Solution A). Dilute 10 ml of Solution A to one litre by distilled water (Solution B). Further dilute 4 ml of Solution B to one litre with acrylontrile (Solution C). Dilute 10, 30, 50, 70, 90 ml of Solution C to 100 ml with acrylonitrile. These solutions are equal to 0·1, 0·3, 0·5, 0·7 and 0·9 ppm of peroxide, respectively. Analyse these standard solutions as given in A-17.3 and plot a curve of absorbance against ppm.

A-17.5 Calculation — Find out ppm of peroxide for the sample from the calibration curve.

A-18. DETERMINATION OF HEAVY METALS (AS Cu)

A-18.1 Apparatus

A-18.1.1 *Spectrophotometer* — Reckman *DB* or equivalent with 10 mm path cells.

A-18.1.2 p*H Meter*

A-18.2 Reagents

A18.2.1 *Perchloric Acid* — 70 percent reagent grade.

A-18.2.2 *Ammonium Citrate Solution* — 20 percent. Dissolve 80 g of reagent grade ammonium citrate in 360 ml of distilled water. Filter if solution is cloudy.

A-18.2.3 *Sodium Diethyldithiocarbamate* — 0·1 percent solution. Dissolve 0·1 g of sodium dithocarbamate in distilled water and make up to 100 ml.

A-18.2.4 *Copper Stock Solution* — 5 microgram/ml. Dissolve 0·196 5 g of reagent grade

$CuSO_4.5\ H_2O$ in distilled water and dilute 100 ml of above solution to 100 0 ml in volumetric flask with distilled water.

A-18.2.5 *Acid Mixture* — Mix 5 parts concentrated sulphuric acid (H_2SO_4) with 2 parts concentrated nitric acid (HNO_3).

A-18.3 Procedure — Transfer 125 ml of sample in 250-ml beaker and evaporate on water bath in fume cupboard. Cool the beaker and add 3 ml of acid mixture. Place beaker on hot plate and allow to boil. Cool and further add 0·2 ml of 70 percent perchloric acid ($HClO_4$). Again place on hot plate and heat to fumes. If the residue is coloured after cooling, repeat acid addition. Transfer the contents to 100-ml beaker and make up the volume to 10 ml. Add 5 ml of 20 percent ammonium citrate and adjust the *p*H to 9·1 ± 0·1 with concentrated ammonium hydroxide (NH_4OH). Transfer quantitatively to 25 ml volumetric flask. Add 1 ml of carbamate solution and dilute to mark. Mix well. Record absorbance using 10 mm path cells at 488 nm. Adjust the zero against distilled water blank.

A-18.4 Preparation of Calibration Curve — Pipette 1, 2, 3, 4 and 5 ml of copper stock solution 5 microgram/ml in separate 100-ml beakers. Add 5 ml of ammonium citrate and adjust the *p*H to 9·1 ± 0·1 with concentrated NH_4OH. Transfer the contents of the above beaker to separate 25 ml volumetric flasks and proceed as given in **A-18.3**. Plot a graph of absorbance against microgram.

A-18.5 Calculation — Read microgram of copper for the absorbance of sample solution from the calibration curve *X* microgram.

$$\text{Cu, ppm} = \frac{X}{V \times 0{\cdot}8}$$

where *V* is the volume of sample in ml.

APPENDIX B

(*Clause* 2.2.1)

DETERMINATION OF HYDROQUINONE CONTENT

B-0. OUTLINE OF THE METHOD

B-0.1 This method of test describes a procedure for determining the hydroquinone content in the range of 1 to 20 ppm. In this procedure, the material is evaporated either by air drying at room temperature or vaccum evaporation at 75°C. The resulting hydroquinone residue is dissolved in water and titrated with dilute standardized ceric acid sulphate solution.

B-1. APPARATUS

B-1.1 Rotary Film Evaporator

B-2. REAGENTS

B-2.1 Standard Solution of Ceric Acid Sulphate (0.002N) — Dissolve 1·096 g of ceric ammonium nitrate $(NH_4)_2 Ce (NO_3)_6$ in 20 ml concentrated sulphuric acid in a 50 ml beaker. Slowly pour the ceric solution, while stirring, into 200 ml of water contained in a 600-ml beaker. When the solution is complete, transfer this mixture to a 1-litre volumetric flask and dilute to mix with water.

B-2.2 Diphenylamine Indicator — Dissolve 0·1 g of diphenylamine in 100 ml concentrated sulphuric acid. Store this solution in a brown bottle.

B-2.3 Standard Hydroquinone Solution — Dissolve 0·2 g of hydroquinone weighed nearest to 0·1 mg in water and dilute to one litre in a volumetric flask. This solution is unstable and should be discarded after one week of normal use.

B-3. PROCEDURE

B-3.1 Pipette 50 ml of acrylonitrile into each of the two 125 ml flasks. Connect the flasks to rotofilm evaporator and apply vacuum. Keep the flask in a hot water bath maintained at 75 ± 5°C. Evaporate the sample to dryness. Do not exceed temperature of 80°C or 30 minutes of evaporation time. Carefully repressure the flask and disconnect. Displace any remaining vapours by carefully purging the flask with dry air/nitrogen. Dissolve the residual hydroquinone in 25 ml water. Add 3 drops of diphenylamine indicator using the same dropper every time. Titrate each solution with 0·002 N ceric acid sulphate to a faint blue point which persists for 15 seconds.

B-3.2 Pipette 10 ml of standard hydroquinone solution (**B-2.3**) into each of two 125 ml Erlenmeyer flasks. Add 3 drops of diphenylamine indicator using the same dropper as used for the test and titrate with 0·002 N ceric acid sulphate solution to a faint blue point which persists for 15 seconds

NOTE — Duplicate titrations should agree within 0·05 ml. Use the average of the two values.

B-4. CALCULATION

$$\text{Hydroquinone content in ppm} = \frac{V \times F}{M} \times 100$$

where

V = volume in ml of ceric acid sulphate solution used to titrate the sample,

$$F = \text{Factor} = \frac{\text{mg of hydroquinone in 10 ml of sample}}{\text{volume in ml of ceric acid sulphate solution required to titrate the standard solution}}, \text{ and}$$

M = mass in g of sample taken.

B-5. REPORT

B-5.1 Report the hydroquinone content nearest to 0·1 ppm.

APPENDIX C

(*Clause* 5.1)

SAMPLING OF ACRYLONITRILE, TECHNICAL

C-1. GENERAL REQUIREMENTS OF SAMPLING

C-1.0 In addition to precautions given in 3 while preparing, storing and handling samples, the following precautions and directions shall be observed.

C-1.1 The persons sampling and testing the material shall wear suitable working clothes, protective eye glasses, rubber gloves and gas mask.

C-1.2 Sampling shall be carried out with the aid of special sampling instrument or clean glass tubes, taking equal subsamples from the lower, middle and upper layers of the product.

C-1.3 The samples shall be placed in clean and dry bottles provided with a ground-glass stopper.

C-1.4 Each sample container shall be sealed air-tight after filling and marked with full identification particulars, such as, the name of the product, the sample number, the date of sampling, lot number, date of manufacture of the materials, etc.

C-2. SCALE OF SAMPLING

C-2.1 Filling of the Material in Tanks — A representative sample shall be taken from each tank. The total volume of the sample taken from each tank shall not be less than 1·5 litre. The sample shall be thoroughly mixed and divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

C-2.1.1 The material in a tank shall be considered as conforming to the requirements of the specification if the test sample meets all the requirements given in the specification.

C-2.2 Packing of Material in Steel Cans and Drums

C-2.2.1 Lot — In a single consignment, all the containers belonging to the same batch of manufacture shall constitute a lot. If the consignment consists of different batches of manufacture, the containers belonging to the same batch of manufacture shall be separated and shall constitute separate lots.

C-2.2.2 For ascertaining the conformity of the material in a lot to the requirements of the specification, tests shall be carried out for each lot separately.

C-2.2.3 The number of containers to be selected (n) for this purpose shall depend on the size of the lot (N) and shall be in accordance with Table 2.

TABLE 2 SCALE OF SAMPLING FOR CONTAINERS

(*Clause* C-2.2.3)

LOT SIZE N (1)	NO. OF CONTAINERS TO BE SELECTED n (2)
Up to 25	3
26 ,, 50	5
51 ,, 100	8
101 ,, 300	13
301 ,, 500	20
501 and above	32

C-2.2.3.1 The containers shall be selected from the lot at random. In order to ensure the randomness of selection, a reference may be made to IS : 4905-1968*. In case this standard is not readily available, following procedure may be adopted:

Starting from any container in the lot, count them as 1, 2, 3.........and so on up to r in one order where r is the integral part of N/n, every r th container thus counted shall be withdrawn till the requisite number of containers is obtained.

C-2.2.4 *Test Sample and Referee Sample*

C-2.2.4.1 From each of the containers selected according to **C-2.2.3**, approximately equal quantity of material shall be taken and thoroughly mixed so as to form a composite sample. The total volume of the composite sample shall be not less than 1·5 litres. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee. While preparing the sample and transferring it to the sample containers, general precautions given in **C-1.0** shall be observed.

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*Methods of random sampling.

C-2.2.4.2 *Referee sample* — The composite sample marked for this purpose shall bear the seals of the purchaser and the supplier. It shall be kept at a place agreed to between the two so as to be used in case of a dispute.

C-2.2.5 *Number of Tests* — Tests for determining all the requirements given in **2** shall be carried out on the composite sample.

C-2.2.6 *Criteria for Conformity* — A lot shall be declared as conforming to the requirements of the specification, if all the test results on the composite sample meet the corresponding requirements given in **2**.